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भारतीय मानक

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Indian Standard

MEASUREMENT OF MERCURY LEVEL IN FLUORESCENT LAMPS

ICS 29.140.30

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BUREAU OF INDIAN STANDARDS
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Electrical Lamps and Their Auxiliaries Sectional Committee, ETD 23

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Electrical Lamps and Their Auxiliaries Sectional Committee had been approved by the Electrotechnical Division Council.

The method of measurement of mercury level covered under this standard is based on atomic absorption spectrophotometer due to ease of operation, cost effectiveness and precession requirements.

The need of formulating this standard has been realized considering manufacturing potential and demand in the country to measure mercury contents available in fluorescent lamps. This may ease safe disposal of fluorescent lamps due to hazardous nature of availability of excess mercury contents.

This standard is based on IEC Doc: 34A/1230/NP 'Measurement of mercury level in fluorescent lamps' issued by the International Electrotechnical Commission (IEC) based on Japanese Standard.

In the formulation of this standard, necessary assistance has been taken from IS 12041 : 1987 'Method for determination of mercury by atomic absorption spectrophotometer'.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MEASUREMENT OF MERCURY LEVEL IN FLUORESCENT LAMPS

1 SCOPE

This standard specifies the quantitative analysis procedure for mercury contents in fluorescent lamps.

2 REFERENCES

The following standards are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
266 : 1993	Sulphuric acid (*third revision*)
1473 : 2004	Methods of chemical analysis of manganese ores (*first revision*)
5760 : 1998	Argon, compressed and liquid — Specification (*second revision*)

3 GENERAL REQUIREMENTS

3.1 Amount of mercury measured in accordance with this standard should be expressed by 2 significant digits.

3.2 Measurements should be repeated 3 times or more, and the average should be calculated.

3.3 Blank test should be done before sample is treated in order to confirm the blank value has no influence to the sample measurement value.

4 SAMPLE PREPARATION

4.1 Mercury in fluorescent lamps exists in three states as followings:

a) Vapor in a lamp;
b) Liquid metal or as an oxygen compound; and
c) Amalgam.

Recently, although some of the lamps are filled with amalgam in order to put mercury in a lamp accurately, there are also many fluorescent lamps filled with metal mercury.

At the determination of total amount of mercury in a fluorescent lamp there is no need to consider about vapour mercury in a lamp, because the amount of vapour mercury is small in view of mercury vapour pressure at room temperature.

The total amount of mercury is determined by measuring the amount of metal mercury, oxidized mercury and amalgam.

Metal mercury and oxidized mercury easily dissolve in nitric acid, however some kinds of amalgams (for example, Ti/Hg, Bi/Pb/Sn/Hg, etc) cannot be dissolved in nitric acid completely. So another acid should be mixed.

This procedure includes a method to dissolve metal mercury and oxidized mercury (*see* **4.1**), and a method to dissolve some kinds of amalgams (*see* **4.2**).

Total amount of mercury is calculated by adding the measured values of metal mercury, oxidized mercury and amalgam.

4.2 Procedure for Dissolving Metal Mercury and Oxidized Mercury in a Fluorescent Lamp

4.2.1 *Instruments*

a) Syringe;
b) Beaker, volumetric flask, pipette;
c) Paper filter, filter glide;
d) File, scissors, hammer, pincher, rubber tube, Ni/Cr wire for heater and a power supply burner for glasswork and glass rod; and
e) Hot plate.

4.2.2 *Reagents*

4.2.2.1 *Nitric acid* — Nitric acid specified in IS 264 or that at least equal there to in quality should be used. In the following procedure nitric acid refers to this.

4.2.2.2 *Water* — Distilled water or ion-exchange water should be used. Water refers to distilled water or ion-exchange water from here onwards.

4.2.2.3 *Nitric acid* (1 + 1) — Same volumes of nitric acid and water are mixed.

4.2.3 *Sampling Procedure*

Fluorescent lamp without amalgam should be treated by method given in **4.2.3.1** or **4.2.3.2**.

Fluorescent lamp with amalgam should be treated by method given in **4.2.3.2**.

4.2.3.1 Mercury decomposition in a lamp bulb, through pouring nitric acid in the lamp bulb.

a) Remove base (only one side) and break the tip of exhaust tube.
b) Pour proper amount of diluted nitric acid into the lamp (for example, 30 ml via 40 W lamp) (*see* Note 1 and Fig. 1);

c) Rinse the inside of lamp thoroughly until no more phosphor comes off. When the solution is spilled, stop the procedure and start from another lamp. Lay the lamp on a hot plate and heat the solution up to 80 to 100°C and keep for 40 min. At this point, mercury in the lamp is completely dissolved.

d) Cut the bulb at the position of about 30 mm from the end of the bulb. The solution is poured into a beaker.

e) Pour 20 ml of diluted nitric acid into the lamp and pour the solution into the beaker mentioned before. After that, rinse the lamp inside of the lamp with about 20 ml of water. The water must be added to the solution mentioned before.

f) Cut the bulb at the position of about 30 mm from the opposite end of the bulb. Rinse the inside of the bulb sufficiently (*see* Note 2).

g) Remove filaments and inner leads from the 2 electrode parts. Rinse 2 electrode parts with 5 ml of nitric acid (1 + 1) and small amount of water. Put them into the beaker mentioned before.

h) The solution is filtered with filter paper and is moved to a volumetric flask. Rinse filter paper with water thoroughly and add water to marked line of a volumetric flask to make the test solution (*see* Note 3).

NOTES

1 By using the pressure difference between atmosphere and inside of the lamp, diluted nitric acid can easily be sucked into the lamp through a rubber tube between the lamp and the beaker, after cut-off the tip (*see* Fig. 2).

2 A negative error could occur when the rinsing of inside of the lamp is poor.

3 This sample solution can be stored for a week. However, diluted test solutions should be measured on the same day.

4.2.3.2 Mercury decomposition in a beaker through breaking the lamp bulb.

a) Remove the base and base cement completely with a knife.

b) Make a scar on near the end of the tube with a file.

c) Make red heated glass rod or Ni/Cr wire contact with the scar in order to make clack and let the lamp bulb leak to atmospheric pressure.

d) Take out amalgams from the inside of the tube. And dissolve them in accordance with the method given in **4.3**.

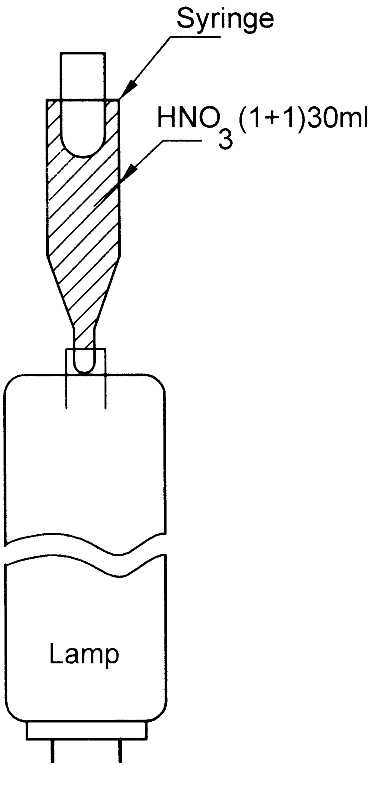


FIG. 1 INJECTION OF NITRIC ACID WITH SYRINGE

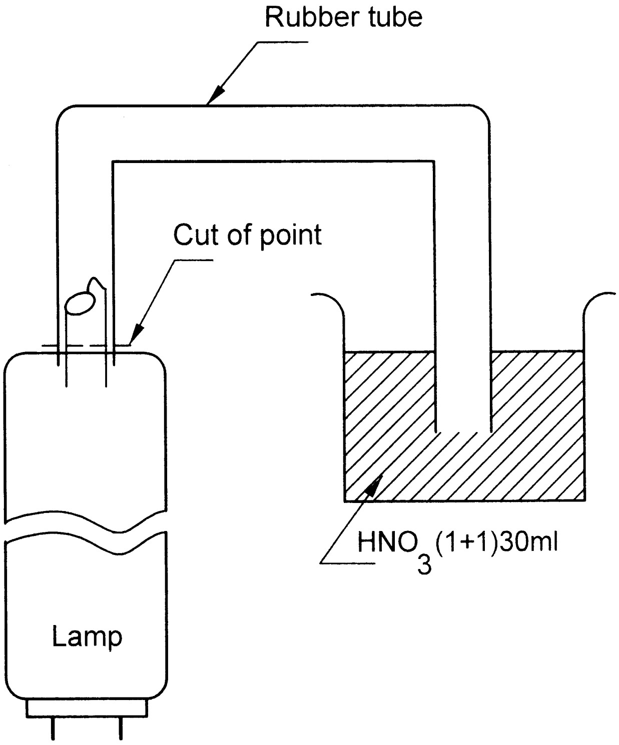


FIG. 2 INJECTION OF NITRIC ACID BY SUCTION

e) Wet all of inside of the bulb with water in order to prevent splash mercury with phosphor when the bulb is broken into pieces. Collect the water in a beaker.

f) Break bulb into pieces inside a thick plastic bag by beating the bulb from outside the plastic bag with a hammer, or break the bulb inside a beaker with pincers. (In order to prevent the plastic bag from breaking, use pincher can be used to make broken pieces smaller instead of beating with hammer repeatedly.)

g) Move pieces of the bulb into a beaker and rinse inside the plastic bag with water (*see* Fig. 3).

h) Add about 30 ml of half diluted nitric acid into the beaker. Wash the pieces of bulb with pipette until no more phosphor coming off from the pieces. Heat the beaker on a hot plate and keep at about 80°C for more than an hour.

j) After cooling to room temperature the sample solution, the solution is filtered with filter paper and is moved to a volumetric flask. Rinse filter paper with water thoroughly and add water to marked line of a volumetric flask to make the test solution (*see* Notes).

NOTES

1 This sample solution can be stored for a week. However, diluted test solutions should be measured on the same day.

2 The amount of dilute depends on the shape of the lamp, since the mercury content of the lamp tube with the amalgam extracted is very small, the amount of dilute should be small as possible.

3 Supernatant solution can be available, when No. 5 filter is used for filtration.

4.3 Decomposition of Mercury Amalgams

4.3.1 *In the Case of Titanium Amalgam*

Example of titanium amalgam equipped in a fluorescent lamp is shown in Fig. 4.

4.3.1.1 *Glassware*

a) Round-bottomed flask, beaker; and
b) Reflux condenser.

4.3.1.2 *Reagent*

a) *Sulphuric acid* — Sulphuric acid specified in IS 266 or that at least equal there to in quality must be used. In the following procedure sulphuric acid refers to this.
b) *Sulphuric acid* (1 + 1) — Same volume of sulphuric acid and water are mixed.
c) *Nitric acid* (1 + 1) — Same volume of nitric acid and water are mixed.

4.3.1.3 *Decomposition of titanium amalgam*

Take out amalgam with metal ring from inside of a lamp (*see* **4.2.3.2**), and put it in a round-bottomed flask. Connect a reflux condenser to a round-bottomed flask

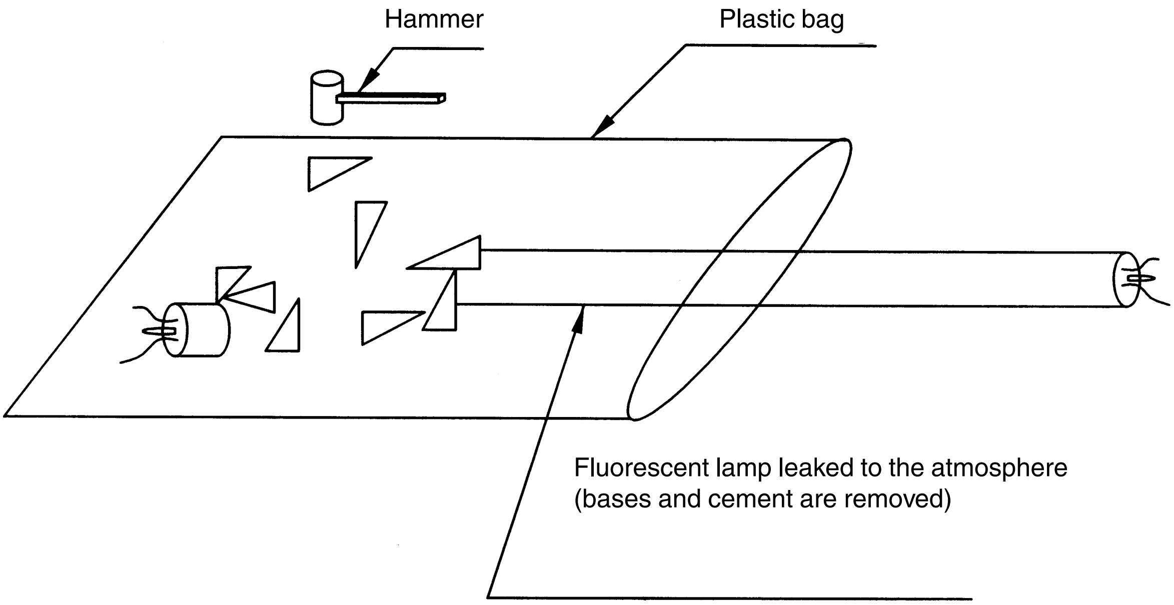

Hammer
Plastic bag
Fluorescent lamp leaked to the atmosphere
(bases and cement are removed)

FIG. 3 EXAMPLE OF THE WAY FOR LAMP DESTRACTION

and cool them by flowing water. Then add 10 ml of sulfuric acid (1 + 1) and heat mildly for about 30 min. Add 5 ml of nitric acid (1 + 1) and heat until some salts formed have completely dissolved and cool it down.

4.3.2 *In the Case of Amalgam Pellet and Amalgam Mesh*

An example of amalgam pellets and amalgam mesh equipped in a fluorescent lamp is show in Fig. 5.

4.3.2.1 *Glassware*

a) Beaker; and
b) Watch glass.

4.3.2.2 *Reagent*

a) *Hydrochloric acid* — Hydrochloric acid specified in IS 265 has to be used; and

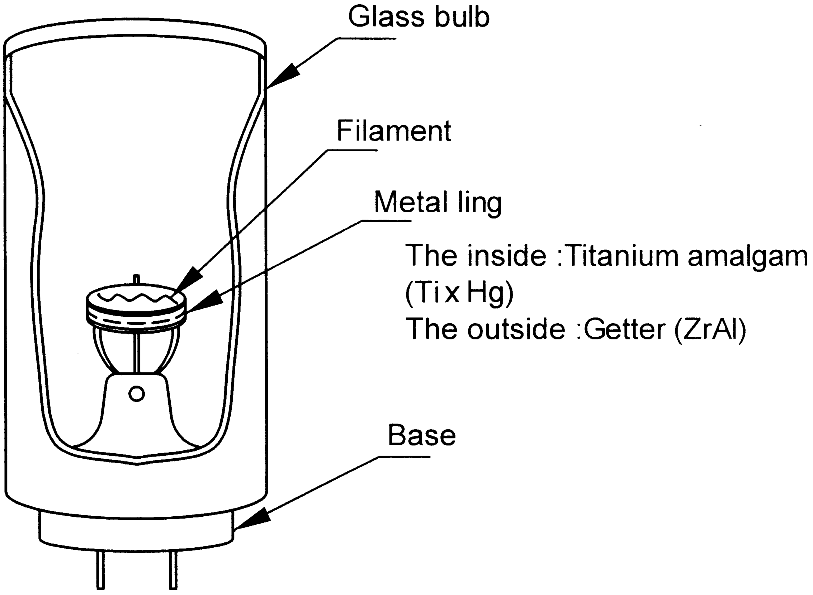


FIG. 4 EXAMPLE OF TITANIUM AMALGAM EQUIPPED IN A FLUORESCENT LAMP ELECTRODE PORTION

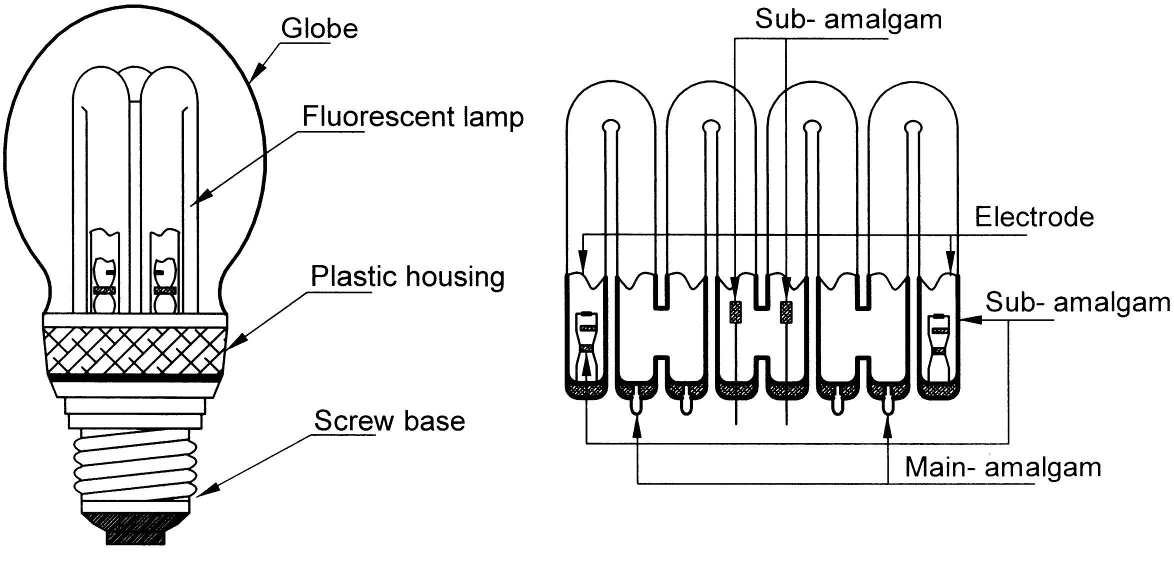


FIG. 5 AN EXAMPLE OF AMALGAM PELLETS AND AMALGAM MESH EQUIPPED IN A FLUORESCENT LAMP

b) *Aqua regia* — Concentric nitric acid is mixed with three times concentric hydrochloric acid.

4.3.2.3 *Decomposition of amalgam pellet*

a) Take out the fluorescent lamp carefully from the plastic housing;

b) Let the fluorescent lamp leak slowly (*see* **5.1.3.2**). Take out the amalgam pellet and the amalgam mesh from the fluorescent lamp;

c) Put them into a beaker and add 10 ml of aqua regia;

d) Heat for 90 min at 80°C; and

e) After cooling to room temperature the sample solution, the solution is filtered with filter paper and is moved to a volumetric flask. Rinse filter paper with water thoroughly and add water to marked line of a volumetric flask to make the test solution.

NOTE — When the solution contains Bi, deposition of Bi may occur in accordance with a drop of acidity. Add aqua regia to make acid concentration more than 10wt percent so that precipitation of salt does not occur.

5 MEASUREMENT

Reducing vapourization atomic absorption spectrometry or ICP atomic emission spectrometry is used.

5.1 Reducing Vaporization Atomic Absorption Spectrometry

5.1.1 *Abstract*

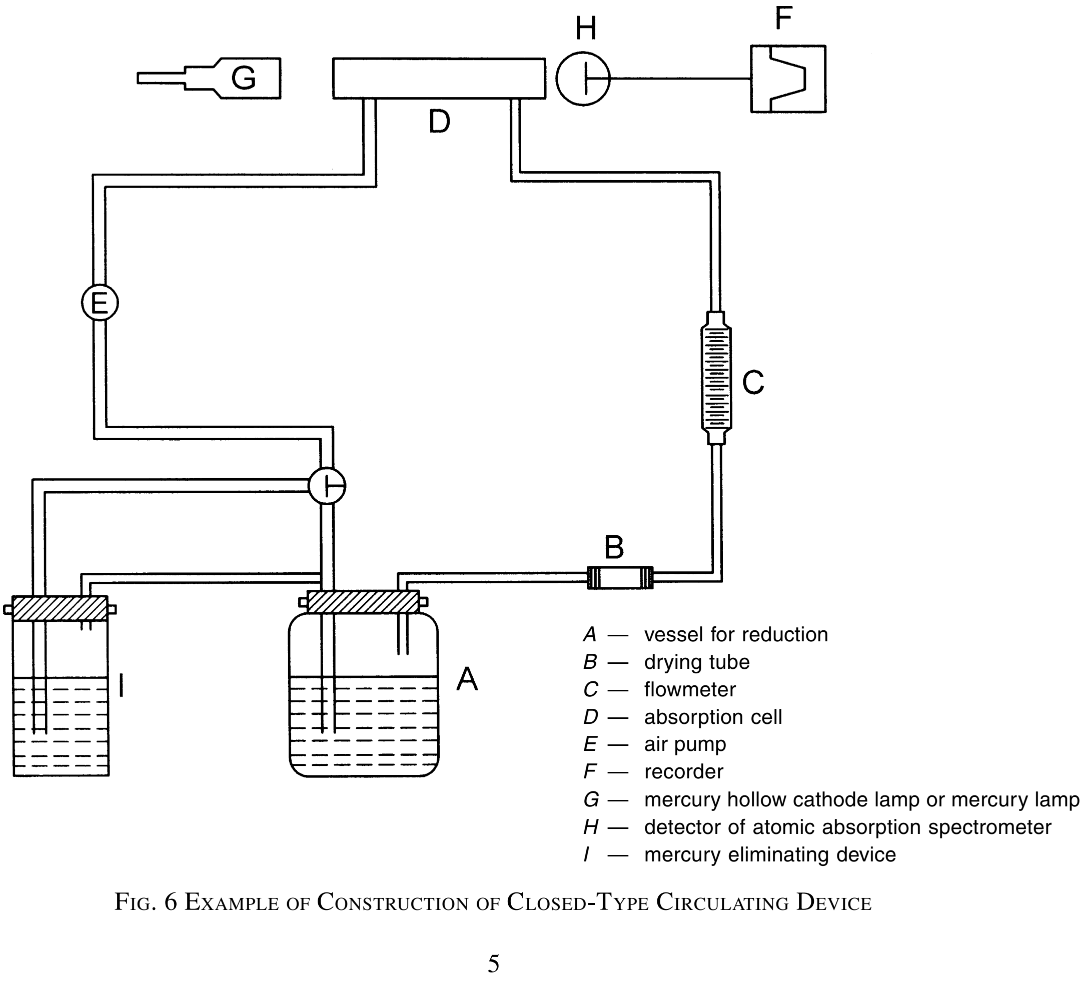
The atomic vapour of mercury is produced with the use of tin (II) chloride as a reducing agent and the mercury vapour is send to the absorption cell. The absorbance of 253.7nm line in UV region is measured to determine the concentration of mercury in the test solution containing mercury and phosphor. Pre-treatment with the use of potassium permanganate can be omitted as the lamp does not contain organic compounds.

5.1.2 *Instrument and Glassware*

a) *Atomic absorption spectrometer* — The instrument which gives atomic absorption signal and precision suitable for an aimed determination range.

b) *Volumetric flask, pipette*

One of atomic absorption spectrometer system diagram (*see* Fig. 6) indicates as following:



A — vessel for reduction
B — drying tube
C — flowmeter
D — absorption cell
E — air pump
F — recorder
G — mercury hollow cathode lamp or mercury lamp
H — detector of atomic absorption spectrometer
I — mercury eliminating device

Fig. 6 Example of Construction of Closed-Type Circulating Device

5.1.3 *Reagent*

a) *Sulphuric acid* (1 + 1) — Same volume of sulphuric acid and water are mixed. (not containing mercury more than 1 μgHg/ml).

b) *Tin (II) chloride solution* — Take 10 g or tin (II) chloride dehydrate. Add 60 ml of sulphuric acid (1 + 20), dissolve it while heating and agitating. After cooling, add water to make total 100 ml. The solution can be stored for a week.

c) *Magnesium perchlorate* — For drying agent.

d) *Mercury standard solution (*1 000 *μgHg/ml)* — For the solution for calibration line, mercury standard solution should be diluted on the day of measurement.

e) *Potassium permanganate solution (5 percent)* — Dissolve 5 g of potassium permanganate specified in IS 1473 in 100 ml of sulphuric acid (1 + 5).

5.1.4 *Operation*

a) Take a suitable amount of sample in a reduction vessel (*see* Note 1).

b) Add sulphuric acid (1 + 1) and water to make appropriate volume (*see* Note 2).

c) Add quickly tin (II) chloride solution, and set the vessel in the apparatus.

d) Run the air pump with the optimum flow rate and read the value indicated at the wavelength of 253.7 nm.

e) Find the amount of mercury on the calibration line, and calculate the concentration of total mercury.

NOTES

1 Take 1 ml or more of the sample solution in order to prevent the error larger.

2 This solution should be measured quickly in order not to make the measurement of mercury content lower than the actual value.

5.1.5 *Calibration Line*

Three or more kinds of solutions which are different in the concentration of mercury, are prepared for calibration line (*see* Fig. 7). Using those solutions, the relation calibration line between indicated value and concentration value is prepared to make a calibration line. By using this calibration graph, the concentration of mercury in the test solution corresponding to the indicated value ratio is obtained.

NOTE — Open-type aerating device can also be used (*see* Fig. 8).

5.2 ICP — Atomic Emission Spectrometry

5.2.1 *Abstract*

After dissolving mercury by using nitric acid or mixed acid (nitric acid and other acid) insoluble components are removed by filtering. Appropriately diluted sample solution is sprayed into plasma and the intensity of atomic emission of mercury is measured.

5.2.2 *Instrument and Glassware*

a) *ICP — Atomic emission spectrometer* — The instrument which gives light emission intensity with suitable sensitivity and stability in aimed measurement and concentration range.

b) *Gas* — For the Ar gas, equivalent in IS 5760 has to be used.

c) *Glassware* — Clean beaker and volumetric flask.

An example of ICP-AES is shown in Fig. 9.

5.2.3 *Reagent*

a) *Water* — The water should be ion exchange water or ion exchange distillation water must be used.

b) *Mercury standard solution* (1 000 *μgHg/ml*) — For the solution for calibration line,

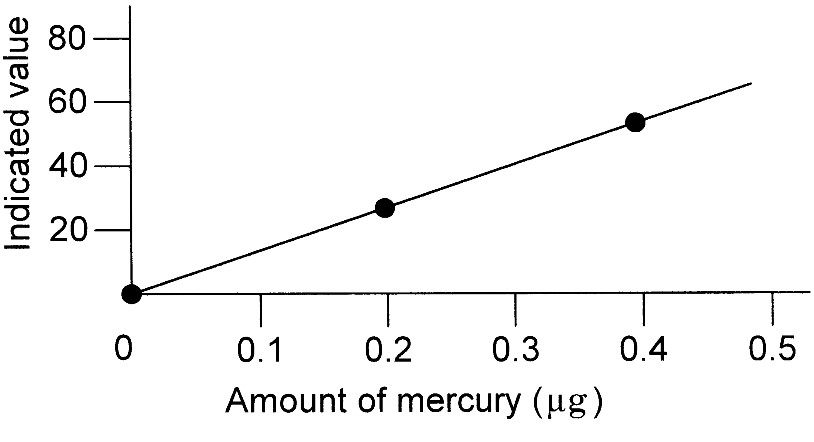
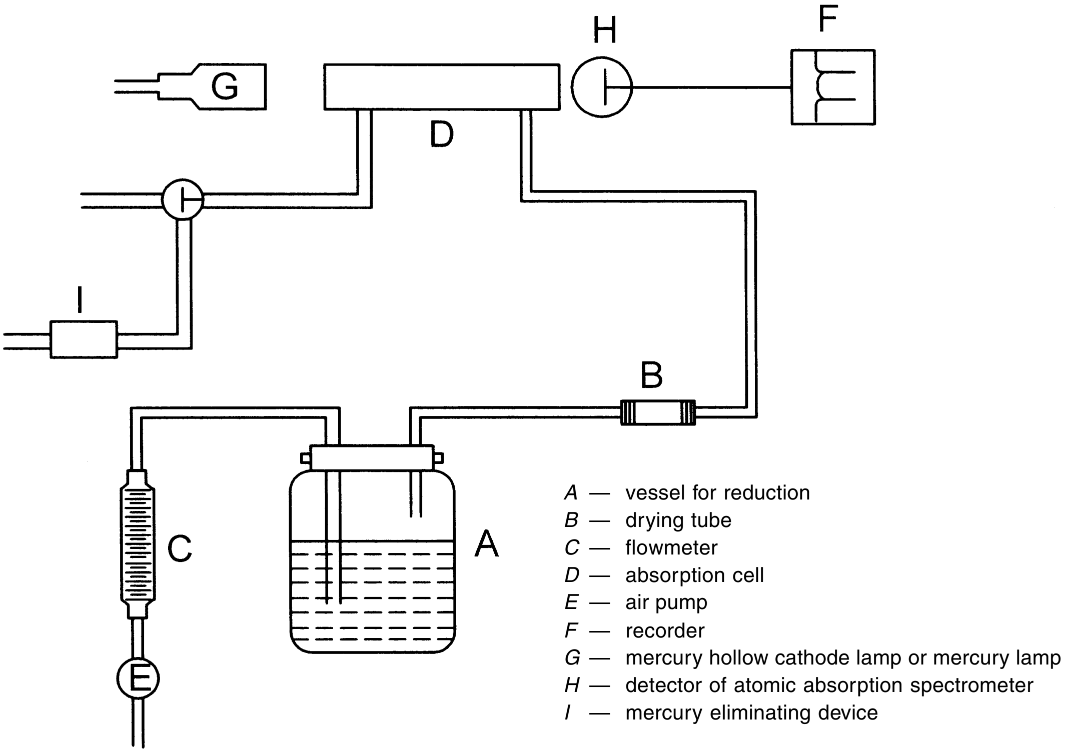


FIG. 7 CALIBRATION LINE



A — vessel for reduction
B — drying tube
C — flowmeter
D — absorption cell
E — air pump
F — recorder
G — mercury hollow cathode lamp or mercury lamp
H — detector of atomic absorption spectrometer
I — mercury eliminating device

FIG. 8 EXAMPLE OF CONSTRUCTION OF OPEN-TYPE AERATING DEVICE

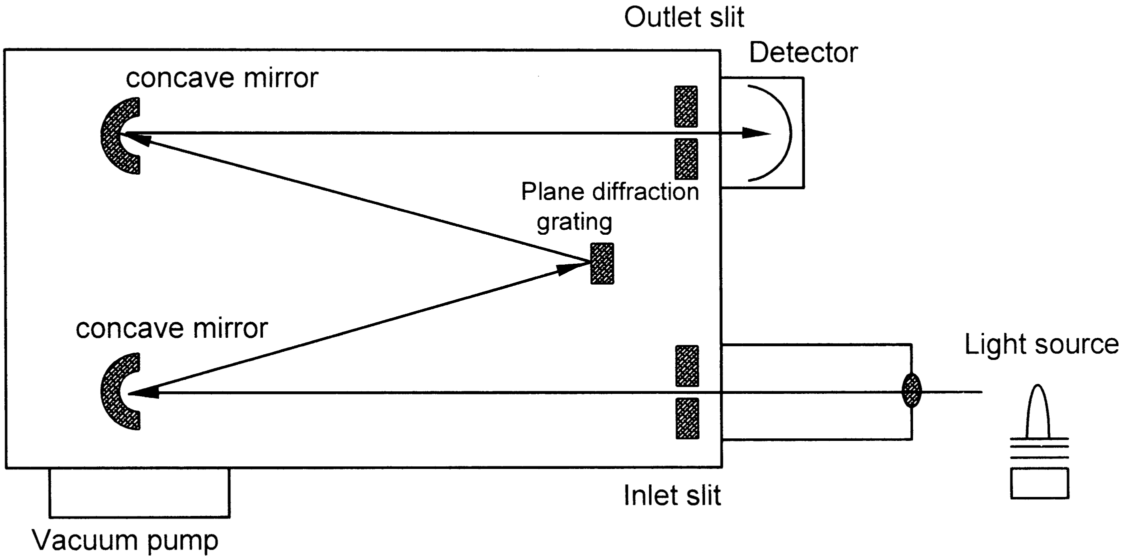


FIG. 9 SPECTROPHOTOMETRY PART USING SEQUENTIAL TYPE SPECTROSCOPE (AN EXAMPLE)

mercury standard solution should be diluted on the day of measurement.

5.2.4 *Setting of Measuring Conditions*

Plasma output, photometric height, flow rate of carrier gas, etc are varied to set the optimized conditions.

There is a possibility of memory effect when low mercury concentration sample is measured after high mercury concentration sample. To avoid memory effect, concentration of mercury in the solution or rinse time with no memory effect should be confirmed.

5.2.5 *Quantitative Analysis*

a) Select the mercury analytical line at wavelength of 194.2 nm for measuring light emission intensity. When spectral interference is found not to exist, the analytical line at 253.7 nm or the other spectral line may be used.

b) As the co-existing elements in the sample solution could have spectral interference on the mercury analytical line, mercury spectral line without spectral interference should be selected by scanning around the mercury analytical line with spraying sample solution into the plasma.

c) To avoid the effect of phosphor matrix standard additional method should be used. Atleast four solutions are sampled by an equal amount from test solution to prepare one kind of solution wherein mercury element is not added and at least three kinds of solutions wherein mercury element is added respectively at different concentration.

d) The solutions are sprayed into plasma and the intensity of atomic emission of mercury is measured. The relation curve between the light emission intensity of mercury element in the test solution is obtained from the intercept of the axis of abscissa (concentration) of Fig. 10. Calculate the amount of mercury in the fluorescent lamp with the following formula:

$$W = A \times C \times V/1\,000$$

where

W = amount of mercury in fluorescent lamp in mg;

A = dilution rate;

C = mercury concentration of test solution in µgHg/ml; and

V = amount of test solution in ml.

NOTES

1 As the line spectrum and continuous spectrum by a plasma composing component and a coexisting element are occasionally superimposed with each other for the analytical line as spectral interference, to select the analytical line with no spectral interference should be recommended. When the spectral interference exists, the degree of the interference calculated as the concentration of mercury should be less than 1/100 of the concentration of mercury of the test solution by using synthetic matrix solution.

2 This method can be applied only to the case of no spectral interference, or where the background and the spectral interference are regularly corrected, and the calibration line between the light emission intensity and the concentration keeps good straightness to the low concentration area.

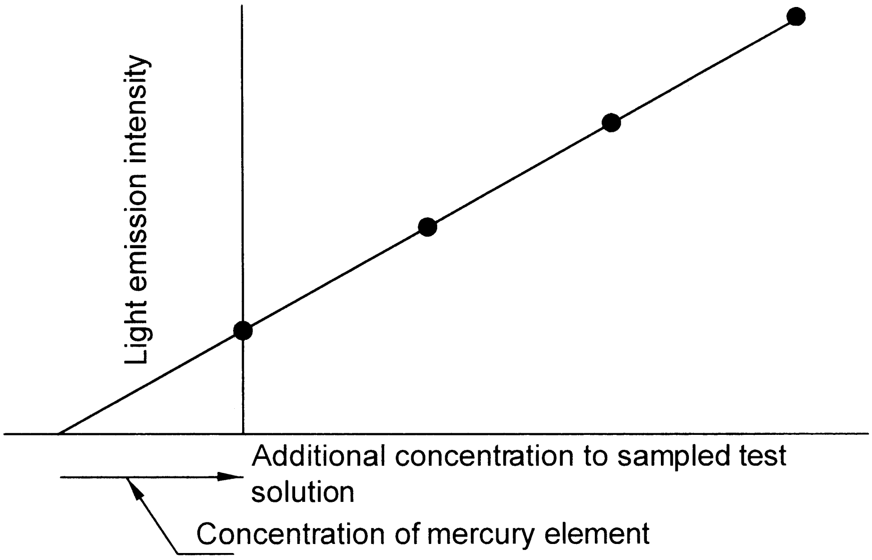


Fig. 10 Method for Obtaining Concentration by Standard Additional Method

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Amendments Issued Since Publication

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